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PROCESSING OF OILY WASTE.
2. LOW–TEMPERATURE PYROLYSIS OF USED M–10DM OIL

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Abstract. The paper presents the results of studying the low-temperature pyrolysis of M-10DM used oil. In the pyrolysis process, pyrolysis oil (pyrocondensate) was obtained as a liquid product. The fractional composition and physicochemical properties of the pyrolysis oil were analyzed. The pyrolysis oil was separated into gasoline fraction, diesel fraction, and a solid residue, the composition and characteristics of which were determined. The pyrolysis oil and distilled fractions were analyzed using X-ray fluorescence and infrared spectroscopy. It is proposed to use the distilled narrow fractions of pyrolysis oil as a raw material to produce motor fuels.
Keywords: oily waste, used oil, pyrolysis, pyrolysis oil, pyrocondensate, gasoline fraction, diesel fraction, residue, char.

1. Introduction

Petroleum oils are an integral component of modern technical infrastructure. They are widely used for lubrication, cooling, and protection of mechanical components and assemblies in vehicles, industrial equipment, and energy systems. However, during operation, oils are subjected to thermal, mechanical, and chemical effects, which lead to degradation of their properties and the formation of used oils (UO).[1-6]

The problem of used oil management is both environmental and economic. On the one hand, UO contain a wide range of polycyclic aromatic hydrocarbons, heavy metals, sulfur, and other toxic impurities. The release of even small amounts of UO into the environment can cause long-term contamination of soil, water resources, and the atmosphere. According to environmental studies, one liter of used oil can contaminate up to one million liters of water, which demonstrates the critical danger of uncontrolled disposal of this waste. On the other hand, used oil is a potentially valuable raw material. It contains a base oil fraction that, after proper purification, can be reused in the production of new lubricants or for energy purposes. At the same time, the use of traditional methods of utilization, such as incineration or disposal, is accompanied by high resource consumption, high emissions of harmful substances, and loss of valuable components.[7-12]

The choice of the optimal utilization technology depends on the volume and composition of UO, economic opportunities, infrastructure availability, and environmental requirements. UO incineration is used mainly for the utilization of oils as an alternative energy source.[13] The process involves the use of special furnaces or boilers adapted for oil incineration or co-incineration with other fuels. The advantages of this utilization method are the use of waste energy, reduced storage of hazardous materials, and the technological simplicity of the process itself. At the same time, the disadvantages of this method are emissions of toxic substances into the atmosphere (sulfur, nitrogen, heavy metal oxides), the need to use complex flue gas cleaning systems, and the loss of valuable hydrocarbon resources that could be reused. The incineration of UO requires strict compliance with environmental regulations, in particular, with regard to controlling emissions of incineration products.[14]

In the modern technology of used oil regeneration, there are a significant number of methods based on physical, chemical, and physicochemical processes. Physical methods (sedimentation, filtration, fuel stripping, rectification) remove mechanical impurities, water, and fuel residues from UO.[15] Physicochemical methods are based on the removal of contaminants by coagulation followed by precipitation, adsorption, or dissolution. Chemical methods of treatment are highly effective for UO with a high degree of contamination, but a significant drawback is the need to introduce a chemically active reagent (acid, alkali, *etc.*), the complexity of separation, and the need to dispose of the aggressive waste.[16,17]

The development of new technologies has led to the emergence of modern alternative methods of carbon-containing waste utilization, among which a special place is occupied by the low-temperature pyrolysis process. The main purpose of this process is the thermal decomposition of carbon-containing waste in an oxygen-free environment to produce gaseous, liquid, and solid products (fuel, carbon materials). Unlike incineration, pyrolysis does not involve burning, but rather the decomposition of complex molecules into simpler products. The advantages of this method are environmental friendliness (minimal emissions into the atmosphere due to the absence of direct burning), energy efficiency (pyrolysis gas can be used to maintain the process temperature), resource recovery (instead of incineration or disposal, carbon-containing waste is converted into useful products), and flexibility (the method is suitable for processing various types of mixed waste).[18] Actually, low-temperature pyrolysis is widely used in the recycling of worn-out car tires, packaging, and plastic waste, and in the creation of facilities for the integrated utilization of municipal solid waste.[19,20] Low-temperature pyrolysis is the technology of the future for processing hard-to-degrade waste. It combines economic benefits, energy efficiency, and environmental safety. Further development of this direction will significantly reduce the anthropogenic impact on the environment and move to a more sustainable circular economy.[21] At the same time, there is little information in the literature on the possibility of processing UO by low-temperature pyrolysis.

In our opinion, the study of such a process for the utilization of used oils would allow to some extent to support the fuel market with an additional number of liquid fuel components obtained from the pyrolysis oil of UO pyrolysis.

The article aims to carry out the process of low-temperature pyrolysis of used M-10DM oil, which is one of the representatives of oily waste, to study the properties of pyrolysis products and propose rational ways of their use.

2. Experimental

2.1. Materials

The starting material used was mineral engine oil of the M-10DM brand, intended for diesel engines. The used oil was selected after the end of the standard engine service life. The main characteristics of the used M-10DM oil are given in Table 1.

2.2. Methods

A comprehensive thermal analysis of used M-10DM oil was performed using a Q-1500 derivatograph of the Paulik-Paulik-Erdey system (IOM, Hungary) connected to a personal computer. The study was performed in dynamic mode at a heating rate of 10 °C/min in an air atmosphere. The weight of each sample was 100 mg. Aluminum oxide was used as a reference material.

Table 1. Characteristics of the used M-10DM mineral motor oil

Index	Value
Viscosity: υ_{50}, mm^2/s	52.12
υ_{100}, mm^2/s	10.78
Density, kg/m^3	879
Acid number, mgKOH/g	2.52
Alkaline number, mgKOH/g	0.39
Water content, %	0.11
Content of mechanical impurities, %	0.054
Coking ability, %	2.26
Ash content, %	0.897
Pour point, °C	–19
Flash point (open cup), °C	218

The low-temperature pyrolysis of used oil was carried out using a laboratory setup consisting of a metal hermetic reactor, a water cooler, and a receiving flask. The temperature in the reactor was controlled by a thermocouple. The used oil was loaded into the reactor and then hermetically sealed with a lid with bolted connections. The refrigerator was connected to the gas outlet tube. After switching on the electric heater, water was circulated through the refrigerator, and the temperature was gradually increased to the required temperature. The pyrolysis oil formed in the refrigerator was collected in a receiving flask. Non-condensed gaseous pyrolysis products (pyrogas) were discharged into the environment. At the end of the process (when the liquid stopped flowing into the receiving flask), the heating was turned off. The amount of pyrolysis oil was determined by weighing the receiving flask before and after the experiment. After the reactor cooled down, it was also weighed to determine the residue yield.

To conduct research and evaluate the possibility of using the pyrolysis oil obtained during the pyrolysis of used oil for the production of motor fuels, the pyrolysis oil was divided into three fractions: IBP-200 °C, 200-350 °C, and the residue above 350 °C. The pyrolysis oil distillation was performed using a classical laboratory setup for fractional distillation of light petroleum products, which included a flask, a flask heater, a water cooler, a spider, and receivers for collecting narrow fractions.

X-ray fluorescence spectral analysis, which was used to determine the elemental composition of both liquid and solid pyrolysis products, was performed on an Elvax Light SDD precision analyzer.

All analyses of the pyrolysis oil, distilled fractions, and the residue were performed following generally accepted standardized methods. The fractional composition of the pyrolysis oil light fractions was determined using an

ARNS apparatus. The iodine number of the liquid fractions was determined by the Margoches method. Additionally, the flash point in closed and open cups, pour point, cloud point, density, and refractive index of pyrolysis oil and the corresponding fractions were determined.

Infrared spectra (FT-IR) of pyrolysis oil and its fractions were obtained using a Spectrum Two spectrometer (PerkinElmer, UK) with a diamond crystal disrupted internal reflection (UATR) attachment. PerkinElmer Spectrum 10 software was used to process and visualize the spectra. Spectra (16 scans per spectrum) were recorded in the mid-infrared range from 4000 to 400 cm^{-1} with a resolution of 1 cm^{-1}.

3. Results and Discussion

To determine the temperature range of thermal decomposition of used oil and study its behavior during heating, derivatographic experiments were conducted (Fig. 1).

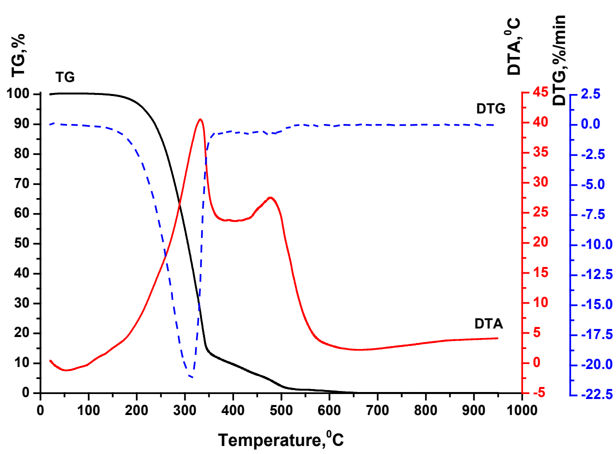

Fig. 1. Derivatogram of a sample of used M-10DM mineral motor oil

Fig. 1 shows two characteristic temperature ranges. In the temperature range from 20 to 387°C, the evaporation of volatile components of the used oil is observed, as well as partial thermo-oxidative degradation, followed by incineration of decomposition products. In this range, the first exothermic peak effect is observed on the DTA curve with a maximum at 331°C. In the range from 387 to 650 °C, the thermal decomposition of oil components and the incineration of pyrolytic residues of the sample continue, accompanied by a second exothermic peak on the DTA curve with a maximum at 479 °C. Taking into account the results of derivatographic studies, a temperature of 420°C was chosen for the low-temperature pyrolysis of used M-10DM oil.

As a result of low-temperature pyrolysis, we obtained the pyrolysis oil - a mixture of hydrocarbons formed as a result of thermal decomposition, a small amount of solid residue in the reactor, and pyrolysis gases. Then, the pyrolysis oil and solid residue were studied, while the pyrolysis gases were not studied, since industrial plants use them for their own needs, burning them to generate heat. The material balance of the pyrolysis process of used M-10DM oil is shown in Table 2.

Pyrolysis oil is a viscous, transparent liquid of light brown color with a characteristic odor of thermal reaction products. An important feature is its high yield, over 90 wt.%. The characteristics of pyrolysis oil are presented in Table 3.

Table 2. Material balance of low-temperature pyrolysis of used M-10DM oil

Raw material and products	Amount, wt.%
Input: Used oil	 100.00
Output: Pyrolysis oil Residue (char) Gas and losses	 91.30 2.00 6.70
Total	100.00

Table 3. Characteristics of pyrolysis oil obtained after pyrolysis of used M-10DM oil

Index	Value
Appearance	Viscous transparent light-brown liquid
Density, kg/m^3	835
Refractive index	1.4728
Sulfur content, wt.%	0.204
Iodine number, $gI_2/100g$	53.4
Pour point, °C	<–18
Flash point In open cup, °C In closed cup, °C	 53 31

The pyrolysis oil obtained after the used oil pyrolysis has a low flash point, which makes it difficult to use as a component of boiler or furnace fuel, and is also characterized by a low pour point. Aromatic hydrocarbons dominate in the structural and group composition of the pyrolysis oil, which is confirmed by the refractive index. The high iodine number indicates the presence of a large amount of unsaturated hydrocarbons. The initial boiling point of pyrolysis oil is slightly higher than that of average oil products and amounts to 65 °C.

For further research, the pyrolysis oil was separated by distillation at atmospheric pressure into a gasoline fraction, a diesel fraction, and a residue. After that, the main operational characteristics of each fraction were determined to assess the possibility of their use in the production of motor fuels.

The characteristics of the gasoline fraction of the pyrolysis oil are shown in Table 4. It was found that the fractional composition of the gasoline fraction (IBP -200 °C) is heavier compared to similar fractions obtained after oil refining. The high content of unsaturated hydrocarbons, confirmed by the iodine number, as well as the increased sulfur content in the gasoline fraction, makes it impossible to use it as a component of commercial motor gasoline without additional technological processing.

Table 4. Characteristics of the gasoline fraction of the pyrolysis oil obtained after pyrolysis of used M-10DM oil

Index	Value
Appearance	Transparent light-yellow liquid
Yield relative to pyrolysis oil, wt.%	13.6
Density, kg/m^3	742
Refractive index	1.4316
Fractional composition, °C:	
IBP	61
10 %	78
50 %	139
90 %	187
EBP	205
Sulfur content, wt.%	0.032
Iodine number, gI_2/100g	74.8

To use the gasoline fraction of the pyrolysis oil as a component of commercial motor gasoline, it is necessary to realize hydrotreating, during which hydrogenation of unsaturated hydrocarbons and sulfur-containing compounds will occur. To increase the octane number of the gasoline fraction, it is advisable to use a catalytic reforming process. This can be performed in a mixture with the straight-run gasoline fraction obtained in the process of oil refining.

The pyrolysis oil fraction with a temperature range of 200-350 °C is characterized by a high flash point (see Table 5), which meets the standard requirements for diesel fuel. The low-temperature properties, such as cloud point and pour point, are sufficient for this fraction to be used in the production of summer diesel fuels. At the same time, the sulfur content exceeds the permissible values, so this fraction can be used in limited quantities as a component of commercial diesel fuel or processed together with the straight-run diesel fraction of oil at hydrotreatment plants.

The residue after distillation of pyrolysis oil with a boiling point above 350 °C is a viscous brown liquid (see Table 6). It contains a significant amount of unsaturated hydrocarbons, as evidenced by the high iodine number, and has an increased sulfur content. Low-temperature properties, in particular the solidification temperature, are a positive characteristic of this residue. At the same time, its high flash point makes it difficult to use as a fuel oil component. However, after additional research, the residue can be used in small doses as an additive to fuel oils. Another promising area is the use of the residue for the manufacture of various types of plastic lubricants, including preservation greases, which also requires further study.

Table 5. Characteristics of the diesel fraction of the pyrolysis oil obtained after pyrolysis of used M-10DM oil

Index	Value
Appearance	Transparent bright-yellow liquid
Yield relative to pyrolysis oil, wt.%	25.1
Density, kg/m^3	825
Refractive index	1.4645
Iodine number, gI_2/100g	62.1
Fractional composition, °C:	
IBP	194
10 %	219
50 %	285
90 %	340
98 %	352
Sulfur content, wt.%	0,053
Cloud point, °C	–12
Pour point, °C	<–18
Flash pont (closed cup), °C	40

Table 6. Characteristics of the residue >350 °C of the pyrolysis oil obtained after pyrolysis of used M-10DM oil

Index	Value
Appearance	Brown liquid
Yield relative to pyrolysis oil, wt.%	63.3
Density, kg/m^3	869
Refractive index	1.4835
Sulfur content, wt.%	0.284
Iodine number, gI_2/100g	44.3
Pour point, °C	–11
Flash point (ope cup), °C	215

The infrared spectra of the used M-10DM oil, the pyrolysis oil, its distilled fractions, and the residue (Fig. 2) show intense absorption bands corresponding to the stretching and bending vibrations of alkanes. In particular, the stretching asymmetric and symmetric vibrations of alkyl CH_2-groups of paraffin are observed at 2919 and 2850 cm^{-1}, respectively, for all samples. Bending vibrations were recorded at 1465 cm^{-1} for all samples. In sample 3, the intensity of vibrations of CH_2-groups is much lower, which can be explained by the shorter length of the carbon chain and the lower molecular weight of compounds in this fraction. At the same time, sample 3 shows a higher intensity of absorption bands corresponding to the vibrations of terminal CH_3 groups: stretching vibrations of CH_3 are recorded at 2953 cm^{-1}, and bending vibrations at

1380 cm⁻¹. This indicates a more branched structure of paraffins in this sample. In addition, the spectrum of the used oil contains absorption bands characteristic of the residues of the anti-aging additive in the range of 1250-1000 cm^{-1}. They are absent after pyrolysis, indicating that these compounds are destroyed during the process.

In all samples, there is no absorption in the range of 3600-3200 cm^{-1}, which indicates the absence of hydroxyl (OH) groups and water. At the same time, all the studied products show absorption bands characteristic of esters and acids, but with different intensities. In particular, in sample 1, an absorption band at 1700 cm^{-1} is recorded, which corresponds to the stretching vibrations of carboxylic acids, while in sample 4, this band is shifted towards longer wavelengths and corresponds to the vibrations of keto and ester groups. In addition, in sample 1, there are absorption bands in the range of 1250-1100 cm^{-1} corresponding to the vibrations of C-O and C(O)-O groups. For the pyrolysis oil and its fractions, the intensity of these bands is much lower. Thus, the pyrolysis resulted in the decomposition of oxygen-containing compounds, as well as esterification reactions, and the resulting compounds are mainly concentrated in sample 4 (fraction 200-350°C).

The main change after pyrolysis is the appearance of unsaturated bonds, in particular, the terminal unsaturated groups $-CH_2{=}CH_2$. In the original sample 1, unsaturated bonds are present in very small amounts. After pyrolysis, absorption bands characteristic of alkenes appear in all samples. In sample 2 and, accordingly, in samples 3-5, the absorption characteristic of arenes at 1642 cm^{-1} is clearly observed. Even sample 5 retains a small content of unsaturated groups, although without end alkyl groups. The highest content of unsaturated groups is observed in sample 3.

Sample 3 contains a predominant number of compounds with unsaturated vinyl, cis-unsaturated, and terminal methylene groups, as well as with benzene and monosubstituted benzene derivatives. Sample 4 has a lower content of unsaturated groups, but a higher proportion of trans-unsaturated groups and aromatic structures, in particular mono- and disubstituted benzene.

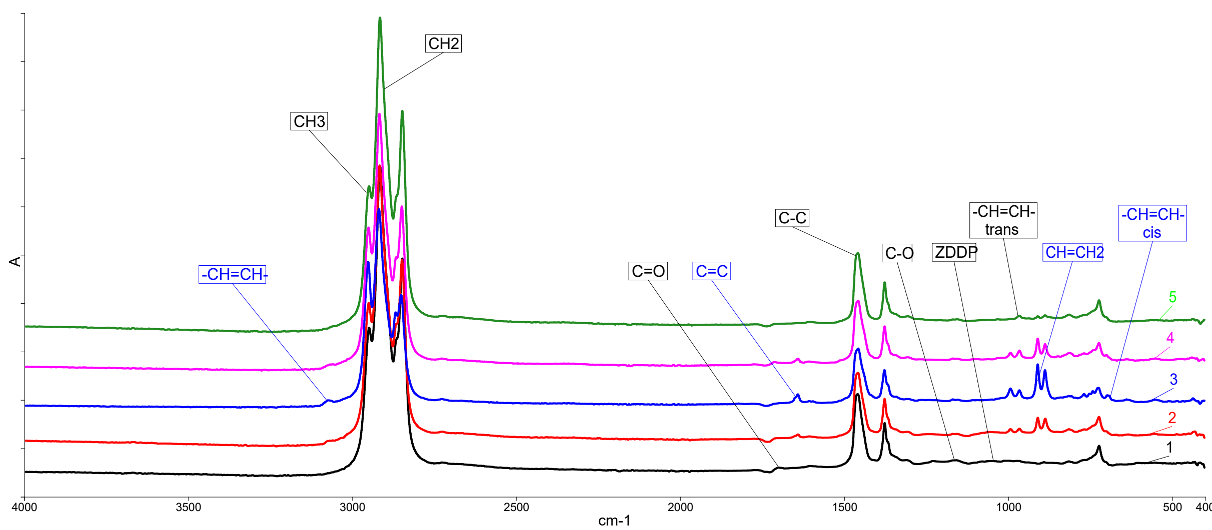

Fig. 2. IR spectra of used M-10DM oil (1); the pyrolysis oil after pyrolysis of used M-10DM oil (2); the gasoline fraction of pyrolysis oil (3); the diesel fraction of pyrolysis oil (4); and the residue after the pyrolysis oil distillation (5)

To determine the concentration of individual chemical elements in the pyrolysis oil, its fractions and the residue, X-ray fluorescence spectral analysis was used (Table 7).

Table 7. Content of individual chemical elements in the pyrolysis oil after pyrolysis of used M-10DM oil

Element	Element content, ppm			
	Pyrolysis oil	Gasoline fraction	Diesel fraction	Residue
1	2	3	4	5
Cu	12.8	10.4	10.7	14.0
Ca	11.5	2.3	8.8	14.2

Table 7. (Continuation) Content of individual chemical elements in the pyrolysis oil after pyrolysis of used M-10DM oil

1	2	3	4	5
Zn	2.0	0.5	0.5	2.9
Fe	< 0.8	< 0.8	< 0.8	< 0.8
Cr	< 1.7	< 1.7	< 1.7	< 1.7
Ni	< 0.3	< 0.3	< 0.3	< 0.3
V	< 0.1	< 0.1	< 0.1	< 0.1
Mn	< 0.1	< 0.1	< 0.1	< 0.1
Ba	< 0.1	< 0.1	< 0.1	< 0.1
Mo	4.8	4.0	4.0	5.2
Pb	< 1.0	< 1.0	< 1.0	< 1.0

The concentration of metals in the pyrolysis oil, its fractions, and the residue was found to be insignificant. Among the detected metals, there are so-called accidental impurities, calcium (Ca) in particular. The heavy metals, such as vanadium (V) and nickel (Ni), are typical of heavy oil fractions and residues. Their absence in pyrolysis oil and its fractions is a positive fact. Studies have shown that, contrary to the generally accepted notion of uneven distribution of metals in oil fractions, metals are distributed quite evenly in the pyrolysis oil, although their highest concentration is in the residue after distillation. The low content of metals in the pyrolysis oil and its fractions is a positive feature, as it facilitates the possibility of their further processing using catalysts.

Table 8. Characteristics of the solid residue (char) obtained during the pyrolysis process of used M-10DM oil

Index	Value
Ash content, wt%	26.4
Water content, wt%	0.25
Volatile content, wt%	6.2
Individual element content, ppm	
Mg	2304.1
Al	1363.3
Si	7352.8
P	79983.4
S	112760.1
K	2183.8
Ca	247279.5
Ti	245.9
Cr	23.9
Mn	23.5
Fe	7905.9
Ni	34.9
Cu	25087.6
Zn	96663.7
Sr	339.6
Pb	432.9

To determine the possible applications of the solid residue obtained during the pyrolysis of used oil, we analyzed its ash content, volatile content, moisture content, and its elemental composition by X-ray fluorescence spectral analysis (Table 8). The ash content of the residue was found to be 26.4 %. The solid residue contains a significant number of different chemical elements (16 identified in total), among which the highest content is accounted for by calcium (Ca), sulfur (S), zinc (Zn), and phosphorus (P). Most of the metals get into the solid residue due to additives contained in the oil: phosphorus, sulfur, zinc indicate the presence of antioxidant and antiwear additives (*e.g.*, DF-11, LANI-317, AntiWear, ZDDP, *etc.*); calcium, manganese (Mn), iron (Fe), strontium (Sr), zirconium (Zr) are components of detergent-dispersant additives (*e.g.*, SK-3); titanium (Ti), molybdenum (Mo), lead (Pb) are present in friction modifiers (*e.g.*, MoS_2, lead naphthenate); molybdenum and lead are also included in corrosion inhibitors, remetallizers and antifriction additives. Fe, Ti, Mn, Mo, and Pb are typical components of any engine that get into motor oil as a result of wear of working surfaces during operation.

The analysis of the pyrolysis material balance (Table 1), together with the data on the content of chemical elements in the pyrolysis oil (Table 7) and solid residue (Table 8) showed that most of the metals present in used oils are converted to solid residue during pyrolysis.

The use of the solid residue (char) obtained during the low-temperature pyrolysis of used oil for the production of solid fuel briquettes is impractical due to its high ash content. Taking into account the chemical composition and properties of this residue, it is advisable to use it as a material for the formation of the lower layer of the road surface.

4. Conclusions

The possibility of recycling used motor oil by low-temperature pyrolysis to obtain liquid products suitable for the production of motor fuels has been established.

The low-temperature pyrolysis of used M-10DM oil at a temperature of 420 °C was carried out, resulting in 91.3 % of pyrolysis oil, 2.0 % of solid residue, and 6.7 % of gaseous pyrolysis products.

The light fractions distilled from the pyrolysis oil can be a raw material for the production of commercial motor fuels, but require additional hydrotreating to reduce the content of unsaturated hydrocarbons and sulfur, as well as to increase the octane number of the gasoline fraction.

The residue formed after pyrolysis oil distillation can be used as a component of fuel oil or as an additive to greases. The solid residue (char) is recommended for use in road construction to form the lower layers of the road surface.

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ПЕРЕРОБКА НАФТОВМІСНИХ ВІДХОДІВ. ПОВІДОМЛЕННЯ 2. НИЗЬКОТЕМПЕРАТУРНИЙ ПІРОЛІЗ ВІДПРАЦЬОВАНОЇ ОЛИВИ М-10ДМ

Анотація. *Представлено результати досліджень низькотемпературного піролізу відпрацьованої оливи М-10ДМ. У процесі проведення піролізу отримано рідкий продукт – піроконденсат. Проведено аналіз фракційного складу та фізико-хімічних властивостей піроконденсату. Здійснено поділ піроконденсату на бензинову, дизельну фракції та твердий залишок, при цьому визначено їхній склад і характеристики. Для піроконденсату та виділених фракцій проведено рентгенофлуоресцентний аналіз, а також ІЧ-спектроскопічні дослідження. Окрім того, досліджено склад і властивості твердого залишку після піролізу відпрацьованих олив. Запропоновано застосовувати виділені вузькі фракції піроконденсату як сировину для виробництва моторних палив.*

Ключові слова: *нафтовмісні відходи, відпрацьована олива, піроліз, піроконденсат, бензинова фракція, дизельна фракція, залишок, твердий залишок.*